

**SULFIDE BY IODOMETRIC METHOD****SM 4500-S2 F-2000 (2011)**

*ADDITIONAL QC REQUIREMENTS FOR THIS METHOD: Certified or Accredited laboratories using this method are assessed to applicable requirements of SM 1020 and SM 4020.*

Facility Name: \_\_\_\_\_ VELAP ID \_\_\_\_\_

Assessor Name: \_\_\_\_\_ Analyst Name: \_\_\_\_\_ Inspection Date \_\_\_\_\_

**Relevant Aspect of Standards****Method  
Reference****Y****N****N/A****Comments**

Records Examined: SOP Number/ Revision/ Date \_\_\_\_\_ Analyst: \_\_\_\_\_

Sample ID: \_\_\_\_\_ Date of Sample Preparation: \_\_\_\_\_ Date of Analysis: \_\_\_\_\_

1. Are samples that are collected for the determination of dissolved sulfides treated in the field with aluminum chloride, allowed to settle and flocculate, and the clear supernatant collected for analysis of dissolved sulfide? (Step may be omitted if sample contains no suspended matter.)

4500-S<sup>2-</sup> B

2. Were samples cooled to ≤6 °C, preserved with zinc acetate plus sodium hydroxide to pH >9, and analyzed within 7 days?

40CFR136.3  
Table 11

3. When samples are pre-treated in the field for the determination of dissolved sulfides, does the chain of custody or other documentation include a record of flocculation step?

2003 NELAC  
5.5.7.3

4. Were 0.0250N standard iodine solutions made by dissolving potassium iodide and iodine in water at rates of 3.2 g iodine/1000 mL water and between 20 and 25 g KI/1000 mL water so that the solution was standardized against 0.0205N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> using starch solution as the indicator?

1.a

5. Were standard sodium thiosulfate titrants prepared at a rate of 1.5 mL 6N NaOH or 0.4 g solid NaOH and 6.205 g Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5H<sub>2</sub>O in 1000 mL distilled water, and were the subsequent solutions standardized with bi-iodate solutions?

1.c

6. Were starch solutions prepared at a rate of 2 g laboratory-grade soluble starch and 0.2 g salicylic acid in 100 mL hot distilled water?

1.d

7. Was sulfide stock standard prepared per SM4500S2-A or purchased? If prepared was stock standard standardized by SM4500S2-F?

SM4500S2-  
A.6.

Notes/Comments:

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
8. Was 2 mL of 6N HCl added to aliquots of iodine solution of sufficient volume to keep iodine color after addition of sample, and were 200 mL aliquots of sample then added?	2.a				
9. Were the combinations of iodine solution and sample then back titrated with sodium thiosulfate solution after the addition of a few drops of starch solution until blue color disappeared?	2.a				
10. Were calculations done correctly?  $\text{mg S}^{2-} / \text{L} = \frac{[(A \times B) - (C \times D)] \times 16,000}{\text{mL sample}}$	3				
11. When samples are pre-treated in the field (flocculation), is the analysis result reported as Dissolved Sulfide?	2003 NELAC 5.5.10.2.h				
Notes/Comments:					